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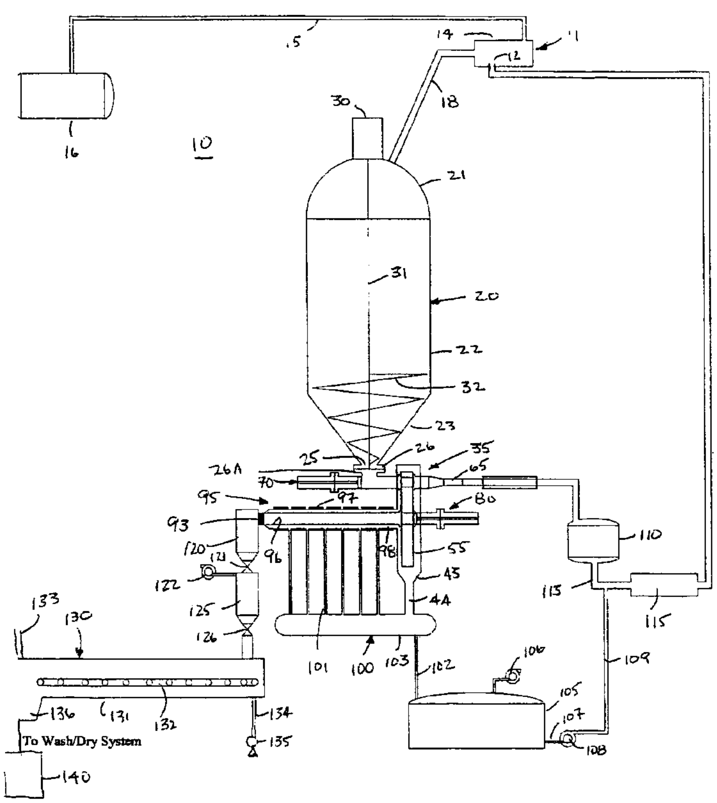

(57) Abstract: A method and apparatus are disclosed for continuously separating a metal powder from a slurry of liquid metal and salt and metal powder. Liquid metal is incrementally expressed to form a cake of metal powder and salt and some metal. Then, the incrementally formed cakes are distilled to vaporize metal leaving metal powder and salt substantially free of liquid metal followed by washing the salt from the metal powder and drying the metal powder. Also disclosed are a variety of products made from the metal powder.

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INDEXING SEPARATION SYSTEM

BACKGROUND OF THE INVENTION

This invention relates to a separation process useful in treating slurries, particularly slurries comprised of metal and non metal particulates such as Ti or Ti alloy powder or particulates, salt, and liquid metal. More particularly, the invention relates to a separation process for a slurry which is produced according to the Armstrong Process as disclosed in each of the three U.S. patents issued thereon, more specifically U.S. patent no. 5,779,761, U.S. patent no. 5,958,106, and U.S. patent no. 6,409,797, the entire disclosures of all three patents being herein incorporated by reference.

Although various metals of controlling the reaction temperature are disclosed in the Armstrong Process, the most commercially advanced method, at the present time, is the use of excess reductant metal, such as sodium, to absorb the heat of reaction during the exothermic reduction of the halide gas, such as titanium tetrachloride to product titanium or a combination of chlorides to produce an alloy. Using an excess of liquid reductant metal, there is produced as reaction products, a powder of the element or alloy to be produced, a particulate salt and excess reductant metal. It should be understood that the scope of this invention is beyond the product of the Armstrong Process and extends to any slurry composed of liquid metal and particulates in which the particulates have to be separated from the liquid metal and thereafter treated. For purposes of brevity only, but not by way of limitation, the description will be in terms of the exothermic reduction of titanium tetrachloride with sodium to produce titanium particles, sodium chloride particles and excess sodium.

During the commercialization of the Armstrong Process, in particular, there is a requirement to handle expeditiously the product from the Armstrong reactor, because the product is formed so quickly the continuous processing thereof has become extremely important. The present invention provides a system and method for handling the product produced by the Armstrong Process such that the Armstrong reactor may be operated continuously to produce 2,000,000,000 pounds per year of elemental metal or alloy while utilizing a single separation vessel and system for the reactor. This is important because it permits the Armstrong reactor to be operated twenty-four hours a day, 7 days a week, economically to produce metal or alloys or any material made by the Armstrong Process as well as applying to handling other slurries as hereinbefore stated.

SUMMARY OF THE INVENTION

Accordingly, an object of the present invention is to provide a continuous method for treating a slurry of liquid metal and particulates.

Another object of the present invention is to provide a continuous method, system and apparatus for separating particulates from a liquid metal slurry containing same.

Yet another object of the present invention is to provide a method of continuously separating a metal powder from a slurry of liquid metal and salt and metal powder, comprising incrementally expressing liquid metal from the slurry to form a cake of metal particulates and salt and some metal, distilling the incrementally formed cakes to vaporize metal leaving metal powder and salt particulates substantially free of liquid metal, washing the salt from the metal powder, and drying the metal powder.

Still another object of the invention is to provide a method of the type set forth comprising introducing a metal halide gas into the liquid phase of a reductant metal to produce a slurry of metal powder and excess reductant metal and a halide salt of the reductant metal, incrementally expressing liquid reductant metal to form a cake, distilling the incrementally formed cakes to vaporize reductant metal leaving metal powder and salt substantially free of reductant metal, passivating the metal powder, washing the salt from the passivated metal powder, and drying the metal powder.

A further object of the present invention is to provide a system of continuously producing metal powder, comprising a reaction chamber for introducing a metal halide gas into the liquid phase of a reductant metal to produce a slurry of metal powder and excess reductant metal and reductant metal halide salt, compression apparatus for incrementally expressing liquid reductant metal to form a cake with some reductant metal, distillation apparatus for heating incrementally formed cakes to separate reductant metal leaving metal powder and salt substantially free of reductant metal,

apparatus contacting the metal powder with oxygen or water to passivate same, apparatus washing the salt from the passivated metal, and apparatus for drying the metal powder.

Yet another object of the present invention is to provide a system of continuously producing metal powder, comprising a vessel containing a slurry of metal powder and liquid metal and a metal halide salt, and having an opening through which slurry flows, compression apparatus for incrementally expressing liquid metal from the slurry to form a cake with some liquid metal, a distillation apparatus for heating incrementally formed cakes to vaporize liquid metal leaving metal powder and salt substantially free of liquid metal, apparatus washing the salt

from the metal powder, and apparatus for drying the metal powder.

A final object of the present invention is to provide a method and system of the type set forth in which the dried metal powder is formed into a product which may be one or more of a solid, a powder injection molded part, a powder metal molded part, a powder metallurgy molded part, a part formed by cold isostatic pressing, a part formed by hot isostatic processing, a densified metal powder product, a product formed from melting the metal powder and forming a solid product therefrom, a product formed by cold spraying the metal powder in a gas jet, a product formed by subjecting the metal powder to a laser, a product formed by spheridizing the metal powder in a plasma, a product formed by foaming and sintering the metal powder, a product formed by rolling the metal powder into a plate and sintering, a product formed by preforming the metal powder onto a mandrel and rolling to form tubes, a product formed by drawing the metal powder, a product formed by extruding the metal powder, and a product formed by treating the metal powder.

These and other objects of the present invention consists of certain novel features and a combination of parts hereinafter fully described, illustrated in the accompanying drawings, and particularly pointed out in the appended claims, it being understood that various changes in the details may be made without departing from the spirit, or sacrificing any of the advantages of the present invention.

BRIEF DESCRIPTION OF THE DRAWINGS

For the purpose of facilitating an understanding of the invention, there is illustrated in the accompanying drawings a preferred embodiment thereof, from an inspection of which, when considered in connection with the following description, the invention, its construction and operation, and many of its advantages should be

readily understood and appreciated.

FIGURE 1 is a schematic view of a system for practicing the method of the present invention;

FIG. 2 is an enlarged schematic representation of the product filtering disc portion of the system illustrated in Fig. 1 shown in longitudinal sectional view;

FIG. 2A is a schematic representation of the product filtering disc of the system illustrated in Fig. 2 with an additional converging or equivalent screw conveyor mechanism;

FIG. 3 is a horizontal cross-sectional view of the vessel illustrated in Fig. 2;

FIG. 4 is an alternate embodiment of the system illustrated in Fig. 1; and

FIG. 5 is a schematic representation of a variety of processes and products made by or from powder separated from slurries according to the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to Figures 1-3, there is disclosed a system 10 for continuously processing a liquid metal slurry containing particulates. In this description, powder and particulates are used interchangeably. More particularly, the system 10 includes a reactor 11 such as, but not limited to the type shown in the Armstrong Process, including a nozzle12 through which liquid metal flows and having a housing 14 surrounding the nozzle. A gas inlet 15 serves to introduce gas from a source 16 thereof into the liquid metal thereby producing an exothermic reaction as described in the referenced Armstrong patents. The product from the exothermic reaction may be a slurry of a liquid reducing metal, such as sodium, having dispersed therein particles of the element or alloy produced, such as titanium or an alloy thereof, and the reaction product from the gas, which may be sodium chloride or combination of chloride salts, as in the case of sodium and titanium tetrachloride. The slurry leaves

the reactor housing 14 through an outlet 18 and is introduced into a receiving vessel 20 having near the top thereof a dome portion 21 and a cylindrical portion 22 terminating in a frustoconical portion 23 having a discharge outlet 25 at the bottom thereof terminating in a circular flange 26. A motor 30 may be mounted at the top of the vessel 20 connected to an output shaft 31 having an agitator 32 at the bottom of the cylindrical portion 22 or the frustoconical portion 23 as illustrated, for a purpose hereinafter set forth.

Indexing filter system 35, as seen particularly in Fig. 2, is in communication with the vessel 20 and more particularly includes a housing 36, having a top 37, a cylindrical side wall 38 provided with opposed upper apertures 39 and opposed lower apertures 41. The housing 36 is also provided with an outlet 43 having a conduit 44 extending therefrom, for a purpose hereinafter set forth.

An index drive 45 includes a motor 46 having an output shaft 47 in communication with a clutch mechanism 48 connected to an axle 49, the end of which rests in a bearing 51. An aperture 49A is provided in the cylindrical wall 38 to accommodate the axle 49.

An indexing disc 55 is rotatably mounted on the axle 49, the disc having a plurality of longitudinally spaced apart chambers 56 therein, six such chambers being shown for purposes of illustration.

A filter 60, preferably but not necessary a metal wedge wire, is positioned in exit conduit 65 and has a collar 61 maintained in sealing contact with the disc 55 through a spring and pin arrangement 62. The chambers 56 in the disc 55 are also in contact with a collar and spring and pin arrangement in communication with an inlet conduit 63 so as to provide a sealing arrangement for each chamber 56 as it rotates about the axle 49. There is, as illustrated in Fig. 2, a T-shaped conduit 66

having flanges and seals 67 connecting the outlet 25 of the vessel 20 to the indexing disc 55, there being provided a sealing flange 26A (Fig. 1) with the usual seals (not shown) to provide a suitable connection between the vessel 20 and the indexing filter system 35.

Compaction ram assembly 70 is mounted to the housing 36 of the indexing filter system 35 and includes a piston rod 71 having mounted thereon a piston 72. The piston rod 71 is surrounded by a bellows seal 73 and is connected at one end to a suitable drive or motor assembly 74 for longitudinal movement toward and away from the chamber 56, as will hereinafter be described.

A discharge ram assembly 80 is mounted to the housing 36 including a similar piston rod 82, bellows seal 83 and drive motor 84 which is similar to the compaction ram assembly 70. In the illustration of Fig. 2, the discharge ram assembly 80 is rotated for clarity but may be positioned anywhere around the housing 36 in which a chamber 56 is positioned during the indexing of the disc 55, as is well understood by any competent engineer. Moreover, the system 10 of the present invention may also include two or more compaction ram assemblies and two or more discharge ram assemblies, each version being a matter of choice of design, well within the skill of the art. The discharge ram assembly 80 further includes, as did the compaction ram assembly 70, collars 86 and springs and retaining pins 87 to ensure a seal between the discharge ram assembly 80, the indexing disc 55 and the outlet conduit 90 which is surrounded by an outer containment tube or conduit 91, for a purpose hereinafter described.

A cake breaker 93 which may be in the form of a stationary grid or flexible members is positioned at the end of the distillation system 95, as will be explained and for a purpose hereinafter described.

A distillation system 95 is in communication with the outlet conduit 90 of the indexing filter system 35 and includes a longitudinally extending conveyor 96 in a container 98 having a heat exchanger 97 in heat exchange relationship therewith. The distillation system 95 is in communication with a condenser assembly 100 by means of one more tubes 101 extending from the container 98 to a condenser container103, it being understood that the condenser container 103 is shown for purposes of illustration as an elongated container but may be of any size or shape as determined. The condenser container 103 is also connected by the conduit 44 to the outlet 43 of the housing 36. A conduit 102 provides communication between the condenser assembly 100 and a liquid metal supply 105 in the form of vessel having connected thereto a distillation vacuum pump 106 and an outlet pipe 107. A pump108 pumps liquid metal from the supply vessel 105 through a conduit 109 to a liquid metal accumulation tank 115. The tank 115 also receives liquid metal from a head tank 110 in communication with the outlet conduit 65 from the indexing filter system 35, the head tank 110 being in communication with the accumulation tank 115 by means of a conduit 113. A heat exchanger 112 may be in heat exchange relationship with the outlet conduit 65 if heat is needed to be added or removed from the liquid metal exiting the indexing filter system 35, as will be explained.

A vessel 120 is positioned in communication with the distillation system 95 and includes a valve 121, the vessel 120 being in communication with a pump 122 which in turn is in communication with a vessel 125 or lock hopper which is also provided with a valve 126 at the bottom thereof. The vessel or lock hopper 125 through valve 126 is in communication with a passivation system 130 which includes a containment vessel 131, a conveyor 132 in communication with a gas inlet conduit 133 and a gas outlet conduit 134 in communication with a pump 135. The

passivation system 130 has an outlet 136, all for a purpose hereinafter set forth.

Operation of the system 10 is as follows. A supply of gas 16 is brought to temperature in a boiler which is meant to be included in the vessel 16 and is transmitted through a conduit or gas inlet pipe 15 into a nozzle 12 through which flows the reductant metal, such as sodium. Sodium is provided from the head tank 110 or supply vessel 105. As is understood, these vessels may be combined into one or may be several, it being within the skill of the art to design the exact combination of parts in the system. Liquid metal pump 105 provides a continuous flow of liquid metal to the nozzle 12 and the amount of liquid metal and gas is adjusted to maintain the temperature in the reactor 11 at a predetermined but generally low temperature of about 400°C. It is understood that various temperatures may be selected as the operating temperature but about 400°C is preferred at the present time. The reaction products in the reactor 11, as previously explained in the Armstrong et al. patents incorporated herein by reference and the illustrated example herein, comprise a slurry of excess sodium, sodium chloride particles and titanium particles. This slurry flows through outlet conduit 18 into the receiving vessel 20. The temperature of the material at this time is still approximately the outlet temperature from the reactor 11 which may be for instance, about 400°C. In the vessel 20, the slurry is stirred in the vessel when the agitator 32 is operated by virtue of the motor 30. The slurry exits the vessel 20 through the discharge outlet 25 thereof into the indexing filter system 35.

Through gravity, the slurry entering the indexing filter system 35 flows into the T-connector 66 and then into inlet conduit 63 through the filter 60 and into the outlet conduit 65. As the liquid sodium flows through the filter 60 which may be for instance, a 125 micron wedge wire filter, the solids concentration increases as liquid

sodium drains. The compaction ram assembly 70 is actuated and the piston 72 drives forwardly into the chamber 56 compressing the material in the conduit 66 thereby expressing liquid metal through the filter 60 until a cake is formed in which most of the liquid metal has been expressed and there remains what could be categorized as wet cake particulate salt and particulate titanium. This cake has sufficient integrity to hold its shape but at the same time still contains some liquid metal. As indicated in the drawing, liquid metal which exits the indexing filter system 35 through the outlet conduit 65 is then recycled to the head tank 110 and moved via the pump 108 back to the nozzle 12 in the reactor 11. After compression by the compaction ram 70 is complete, the motor therefor 74 withdraws the piston 72 and the indexing disc 55 is rotated by the index drive mechanism 45 so as to advance the next chamber 56 into position for another actuation. As is seen from the drawing, because the inlet conduit 66 is connected via gravity to the vessel 20, as soon as the compaction ram 70 withdraws more slurry enters the system. As the disc 55 is rotated as soon as the ram is withdrawn, no slurry material enters the chamber 56 after compaction until the next chamber is in alignment with the compaction ram 70, at which time the aligned chamber 56 fills with the slurry and is thereafter compacted or compressed.

The discharge ram assembly 80 is in alignment or in registry with another chamber 56 of the indexing disc 55 and when the chamber 56 which has the compressed or compacted material therein is in alignment with the discharge ram 80 the piston 82 moves the cake in the chamber 56 into the distillation system 95.

As seen from Fig. 2, there are a plurality of seals 61 and 86 which contain the liquid metal in the appropriate conduit and in its restricted path. However, seals are not necessarily perfect in the real world and although the sealing mechanism in the

conduits are intended to provide a seal for the liquid metal, some inevitably may escape and is collected within the housing 36 and flows out of the outlet 43 and conduit 44 into the condenser assembly 100 for further recycle, as will be explained. Although illustrated with one compaction ram assembly 70 and one discharge ram assembly 80, it is well within the skill of the art to include more than one compaction and/or discharge ram assembly 70, 80.

In the distillation system 95, the particulates from the cake are heated by virtue of the heat exchanger 97, which may be via conduction, convection, induction heating or any other suitable commercial method of heating the powder or particulates moved by the conveyor 96 through the cake breaker 93 to the vessel 120. The cake breaker 93 is shown schematically and may be a fixed series of wires or a variety of other mechanical mechanisms which breaks a compacted particulate matter into a loose friable material. Liquid metal which is vaporized in the distillation system 95 is collection and transmitted via conduits 101 into the condenser assembly 100 and container 103 which is maintained at a sufficiently low temperature to condense the liquid metal vapor into a liquid which is transmitted to storage, such as in head tank 110. As previously described, eventually the liquid metal in the tanks 105 and 110 is recirculated via the pump 108 to the reactor 11 and more particularly the nozzle 12. A valve 121 exists between the vessel 120 and the vessel or lock hopper 125. In addition, pump 122 is in communication with the vessel or lock hopper 125 to ensure no vapors back-up into the system 10 and to evacuate the system if it is necessary to isolate the lock hopper 125 by actuation of the valves 121 and 126. From the lock hopper 125 granular material is transmitted to the passivation system 130 and more particularly to a conveyor 132 which moves within a containment vessel 131 while a passivating gas or liquid is provided through

the gas or water inlet or conduit 133. As illustrated, the particulates move in countercurrent relation to the passivation material but may not be required to do so. Preferably, the passivation fluid is a gas containing a small percentage such as 0.2% by volume oxygen and an inert gas such as argon. The passivated material then moves through outlet 136 to a wash and dry system 140. A pump 135 and conduit 134 exhaust the passivating fluid and recycle same, if desired.

Referring now to Fig. 4, there is shown an alternate embodiment of the present invention in which like numbers have been applied to like parts. The principal difference in the embodiment of Fig. 4 is that the indexing disc 55 as well as the indexing filter system 35 is arranged horizontally instead of vertically so that as slurry drains from the bottom of the vessel 20, liquid reducing metal drains through the filter 60 and a compaction occurs after rotation of the indexing disc 55. Thereafter upon further rotation, the discharge ram assembly 80 is actuated to move the cake into the distillation system 95. Other than the positioning of the indexing disc 55, requiring an indexing between recovering slurry and compaction , the operation of the two systems is identical. Additionally, an advantage of the indexing disc 55 and associated mechanism is that the vessel 20 as well as the conditions therein is isolated from the downstream operations such as the distillation chamber 95 and the passivation station 130. The significance of this is that the conditions in the vessel 20 may be inert or under vacuum or at a specific temperature while the conditions in the distillation station or chamber 95 and/or the passivation station or chamber 130 may be completely different without affecting the upstream conditions in the vessel 20. This isolation of vessel 20 from all downstream conditions provides a significant advantage for the Armstrong process.

Referring now to Fig. 2A, there is a shown an alternate embodiment of the present invention in which a variable pitch converging screw conveyor 230 is positioned intermediate the indexing disc 55 and the distillation system 95 to convey solids or semi-solids between the indexing disc 55 and the distillation system 95. The variable pitch converging screw conveyor 230 shown in Fig. 2A includes a housing 90 (the same as previously described) through which passes a shaft 231 having screw flights 232 positioned thereon, in a well known manner. The screw flights 232 may be electrically heated or may be configured to accept a heat exchange fluid flowing therethrough, such as liquid alkali metal or more specifically sodium when used in The Armstrong Process as previously described. An advantage of using a variable pitch converging screw conveyor 230 is that another seal is then maintained between the vessel 20 and the downstream treatment apparatus by the compaction of solids into continuously diminishing volume between adjacent screw flights 32. A variety of mechanical equivalents of the variable pitch converging screw conveyor 230 may be substituted herein and are within the skill of the art and are incorporated in this invention.

On the assumption that a single Armstrong reactor can produce 2,000,000 pounds per year of product, such as titanium or a titanium alloy such as titanium, 6% aluminum and 4% vanadium, the chambers 56 would be 10 inches in diameter and 6 inches in length. Preferably there are 6 such chambers in each disc 55 based calculations that the slurry and/or gel produced in the Armstrong Process which is approximately 22-23% solids by weight. The indexing disc 55 will be indexed approximately every 11 seconds based on the above-described chambers. Different volume chambers or number of chambers will require different indexing times, but this is well within the skill of the art. Under the present example, the material in the

chamber 56 will be compressed by a factor of 4 so that the cake ejected by the compaction ram assembly 80 will be about 1.5 inches thick and have a solids composition between about 64 and 65 percent by weight.

Referring now to Fig. 5, there is disclosed a schematic representation of the various processes and products made in accordance with the present invention. The reduction box is the Armstrong Process in which a exothermic reduction occurs controlled by the use of constituents taught in the above-mentioned and incorporated Armstrong et al patents. The separation is as previously described herein along with the passivation. The passivated material then is transmitted to a wash and dry assembly 140 in which the salt product, in this specific example, sodium chloride, is removed from the product particulates, in the example titanium or titanium alloy powder.

Referring to Fig. 5, the schematic shows that the powder may be melted to form an ingot or other solid product by a variety of methods such as casting or transmitted to a powder metallurgy process which includes, but is not limited to, for instance isostatic cold processing, hot isostatic processing using pressure to densify the metal powder into a predetermined shape and density. The product may also be produced by cold spraying the metal powder in a gas jet or subjecting the metal powder to a laser or spheridizing the metal powder by plasma. The metal powder may be formed into a foam and thereafter pressed and sintered to form a stable metal foam as is well known in the art. The powder may be pressed onto a mandrel and thereafter rolled into a thin wall tube. Moreover, powder product may be formed by drawing or extruding the metal powder. In the event that product morphology such as the packing fraction, mean size or size distribution needs to be altered, attriting mechanism may be used to change the morphology of the powder, including

the packing fraction or reducing the overall size distribution of the powder.

All these methods of producing product as well as the products formed thereby when combined with the present separation process are included in the present invention.

While there has been disclosed what is considered to be the preferred embodiment of the present invention, it is understood that various changes in the details may be made without departing from the spirit, or sacrificing any of the advantages of the present invention.

WHAT IS CLAIMED IS:

1. A method of continuously separating a metal powder from a slurry of liquid metal and salt and metal powder, comprising incrementally expressing liquid metal to form a cake of metal powder and salt and some metal, distilling the incrementally formed cakes to vaporize metal leaving metal powder and salt substantially free of liquid metal, washing the salt from the metal powder, and drying the metal powder.

2. The method of claim 1, wherein the cake is formed by filling a chamber terminated by a filter.

3. The method of claim 2, wherein a gel is formed against the filter.

4. The method of claim 3, wherein there are a plurality of chambers filled seriatim.

5. The method of claim 1, wherein the incrementally formed cakes are introduced into an elongated distillation chamber.

6. The method of claim 4, wherein the elongated distillation chambers are disposed generally horizontally.

7. The method of claim 1 wherein the metal powder is passivated after the cakes are distilled to be substantially free of the liquid metal.

8. The method of claim 1 wherein the liquid metal is an alkali or alkaline earth metal, alloys or mixtures thereof

9. The method of claim 1 wherein the metal powder is Ti or an alloy thereof.

10. The method of claim 4, wherein the metal powder is Ti or an alloy thereof, and the liquid metal is an alkali or alkaline earth metal, alloys or mixtures thereof .

11. The method of claim 10, wherein the Ti alloy is 6% Al and 4% V with the reminder being substantially Ti.

12. The method of claim 1 wherein the salt is a halide.

13. The method of claim 10, wherein the metal powder has Fe present in an amount less than about 0.30% by weight, oxygen present in an amount less than about 0.25% by weight, nitrogen present in an amount less than about 0.03% by weight, carbon present in an amount less than about 0.1% by weight, and hydrogen present in an amount less than 0.015% by weight.

14. A solid formed from the metal powder made from the method of claim 1

15. A product made from the method of claim 1

16. A product made from the method of claim 13.

17. A product made from the solid of claim 14.

18. The method of claim 1, and further comprising powder injection molding the metal powder to form a product.

19. The method of claim 1, and further comprising forming a product by powder metallurgy with the metal powder.

20. The method of claim 1, and further comprising forming a product by cold isostatic processing the metal powder.

21. The method of claim 1, and further comprising forming a product by hot isostatic processing the metal powder.

22. The method of claim 1, and further comprising forming a product by molding the metal powder into a predetermined shape and exerting sufficient pressure to densify the metal powder.

23. The method of claim 1, and further comprising forming a product by melting the metal powder and forming a solid product therefrom.

24. The method of claim 1, and further comprising forming a product by cold spraying the metal powder in a gas jet.

25. The method of claim 1, and further comprising forming a product by subjecting the metal powder to a laser.

26. The method of claim 1, and further comprising forming a product by spheridizing the metal powder in a plasma.

27. The method of claim 1, and further comprising forming a product by forming foam from pressing and sintering the metal powder.

28. The method of claim 1, and further comprising forming a product by rolling the metal powder into plate and sintering.

29. The method of claim 1, and further comprising forming a product by preforming the metal powder onto a mandrel and rolling into a thinner wall tube.

30. The method of claim 1, and further comprising forming a product by drawing the metal powder.

31. The method of claim 1, and further comprising forming a product by extruding the metal powder.

32. The method of claim 1, and further comprising forming a product by attriting the metal powder to provide one or more of increasing the packing fraction, reducing the mean size or reducing the size distribution.

33. A method of continuously producing metal powder, comprising introducing a metal halide gas into the liquid phase of a reductant metal to produce a slurry of metal powder and excess reductant metal and a halide salt of the reductant metal, incrementally expressing liquid reductant metal to form a cake, distilling the incrementally formed cakes to vaporize reductant metal leaving metal powder and salt substantially free of reductant metal, passivating the metal powder, washing the salt from the passivated metal powder, and drying the metal powder.

34. The method of claim 33, wherein the reductant metal is an alkali or alkaline earth metal, alloys or mixtures thereof.

35. The method of claim 34, wherein the halide is a chloride.

36. The method of claim 35, wherein the metal powder is Ti or a Ti alloy.

37. The method of claim 36, wherein a gel is formed from the slurry before the cake is formed.

38. The method of claim 33, wherein the incrementally formed cakes are introduced into an elongated distillation chamber .

39. The method of claim 38, wherein the cake is formed in a rotatable chamber in communication in one position with the slurry and in another position with the distillation chamber.

40. The method of claim 39, wherein the rotatable chamber is in an indexing disk mounted for rotation about a substantially horizontal axis and the distillation chamber is positioned substantially horizontally.

41. The method of claim 40, wherein there are plurality chambers circumferentially positioned around the indexing disk.

42. The method of claim 41, wherein a horizontally disposed ram compresses the slurry to form a cake and a horizontally disposed ram transports the cake from the rotatable chamber to the distillation chamber.

43. The method of claim 42, wherein the passivating is accomplished with oxygen or water.

44. The method of claim 43, wherein the metal powder is Ti or a Ti alloy.

45. The method of claim 44, and further comprising forming the Ti or Ti alloy powder into a solid.

46. The method of claim 33, wherein the metal powder is Ti or a Ti alloy and is formed into a product by powder injection molding.

47. The method of claim 39, and further comprising forming the metal powder into a solid of Ti of a Ti alloy.

48. A system of continuously producing metal powder, comprising a vessel containing a slurry of metal powder and liquid metal and a metal halide salt, and having an opening through which slurry flows, compression apparatus for incrementally expressing liquid metal from the slurry to form a cake with some liquid metal, a distillation apparatus for heating incrementally formed cakes to vaporize liquid metal leaving metal powder and salt substantially free of liquid metal, apparatus washing the salt from the metal powder, and apparatus for drying the metal powder.

49. The system of claim 48, and further comprising a plurality of chambers for receiving slurry and discharging cake.

50. The system of claim 49, wherein the chambers are circumferentially spaced around a rotatable disk.

51. The system of claim 50, wherein said compression apparatus includes a ram for compressing the slurry against a filter to form a cake.

52. The system of claim 51 and further including a ram for transporting the cake to said distillation apparatus.

53. The system of claim 52, and further including a conveyor for transporting the distilled cakes to said washing apparatus.

54. The system of claim 53, and further including an enclosed conveyor for passing the distilled cake in contact with a gas containing oxygen to passivate the metal powder in the cake.

55. The system of claim 54, wherein the metal powder is Ti or a Ti alloy.

56. The system of claim 55, wherein the Ti alloy contains 6% Al and 4% V.

57. The system of claim 55, and further comprising apparatus for converting the Ti or Ti alloy powder into a solid.

58. A system of continuously producing metal powder, comprising a reaction chamber for introducing a metal halide gas into the liquid phase of a reductant metal to produce a slurry of metal powder and excess reductant metal and reductant metal halide salt, compression apparatus for incrementally expressing liquid reductant metal to form a cake with some reductant metal, distillation apparatus for heating incrementally formed cakes to separate reductant metal leaving metal powder and salt substantially free of reductant metal, apparatus contacting the metal powder with oxygen or water to passivate same, apparatus washing the salt from the passivated metal, and apparatus for drying the metal powder.

59. The system of claim 58, wherein a motor is operably connected to said compression apparatus for indexing a portion thereof.

60. The system of claim 59, wherein said compression apparatus further includes a rotatable disk operably connected to said motor and having a plurality of circumferentially spaced apart chambers each being aligned in one position with a filter and in another position with said distillation apparatus.

61. The system of claim 60, wherein said compression apparatus includes a ram for compressing the slurry against said filter to form a cake.

62. The system of claim 61, and further including a ram for transporting the cake to said distillation apparatus.

63. The system of claim 62, wherein said rotatable disk is positioned to rotate about a substantially horizontal axis and said ram for compression and said ram for transporting are positioned substantially horizontally.

64. The system of claim 63, wherein the filter is stationary.

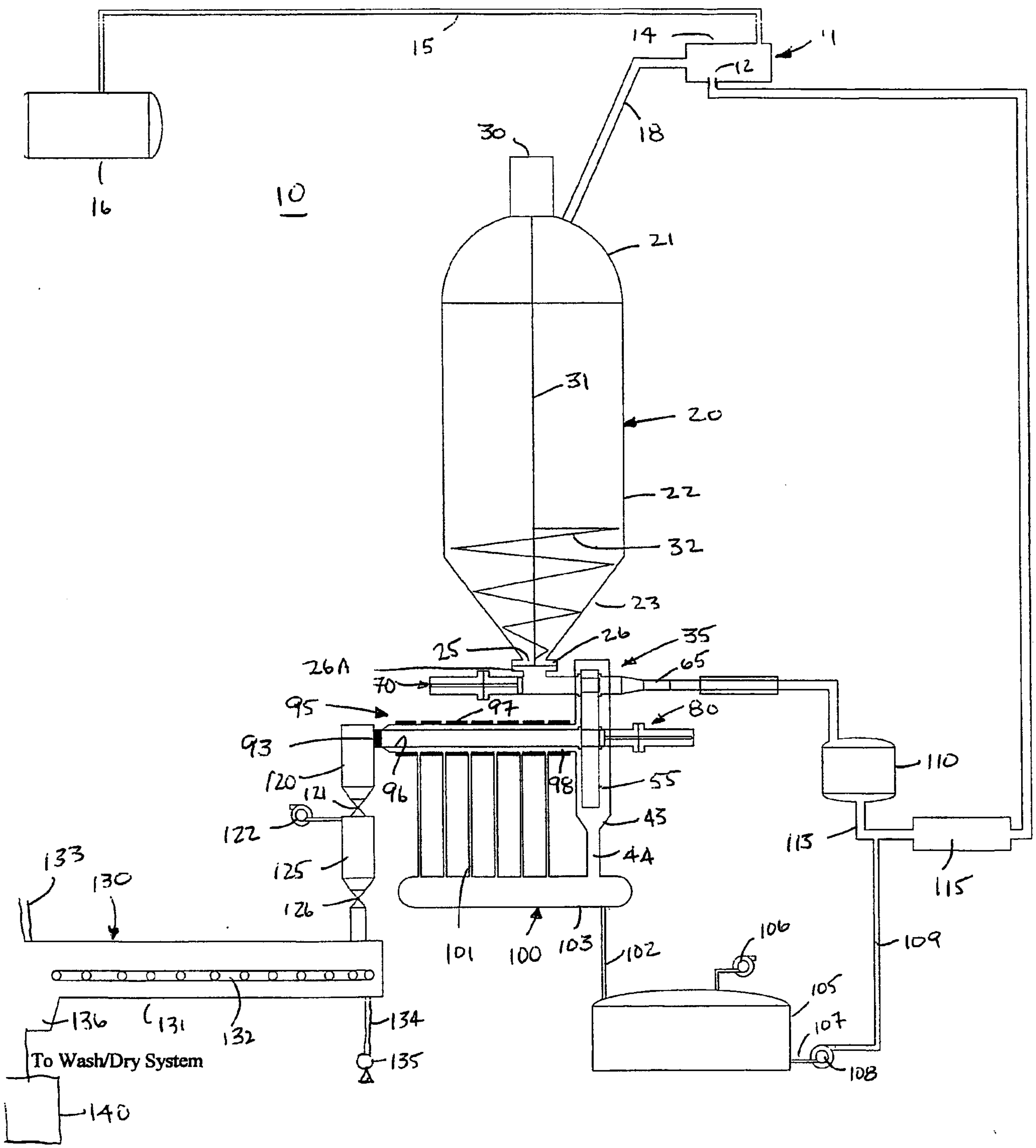
65. The system of claim 64, and further comprising a collection vessel in communication with said compression apparatus and said distillation apparatus to recover reductant metal.

66. The system of claim 65, wherein the reductant metal is Na and the metal halide gas is $TiCl_4$ or includes $TiCl_4$.

67. The method of claim 1, wherein the cake of metal powder and salt and some metal forms a substantially gas-tight seal.

68. The system of claim 48, wherein said compression apparatus forms a seal with the cake therein to isolate said vessel containing the slurry from said distillation apparatus.

Figure 1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To Wash/Dry System
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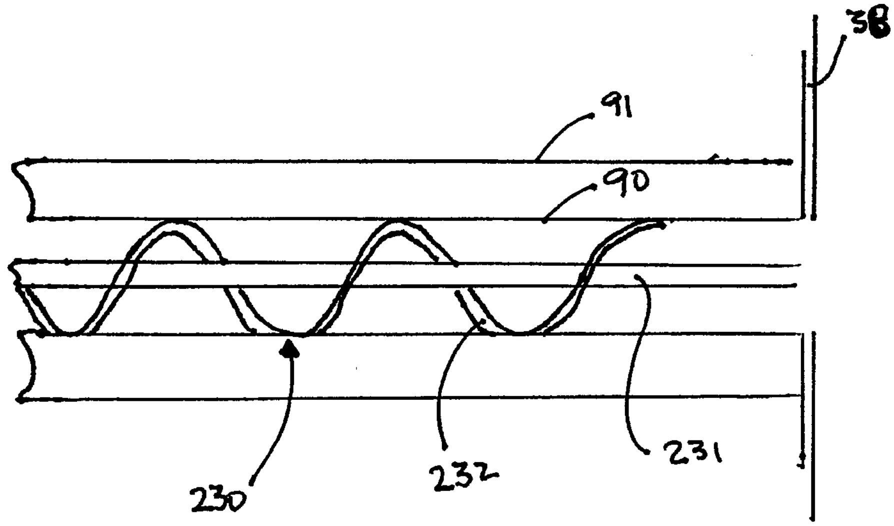


FIGURE 2A

Figure 2

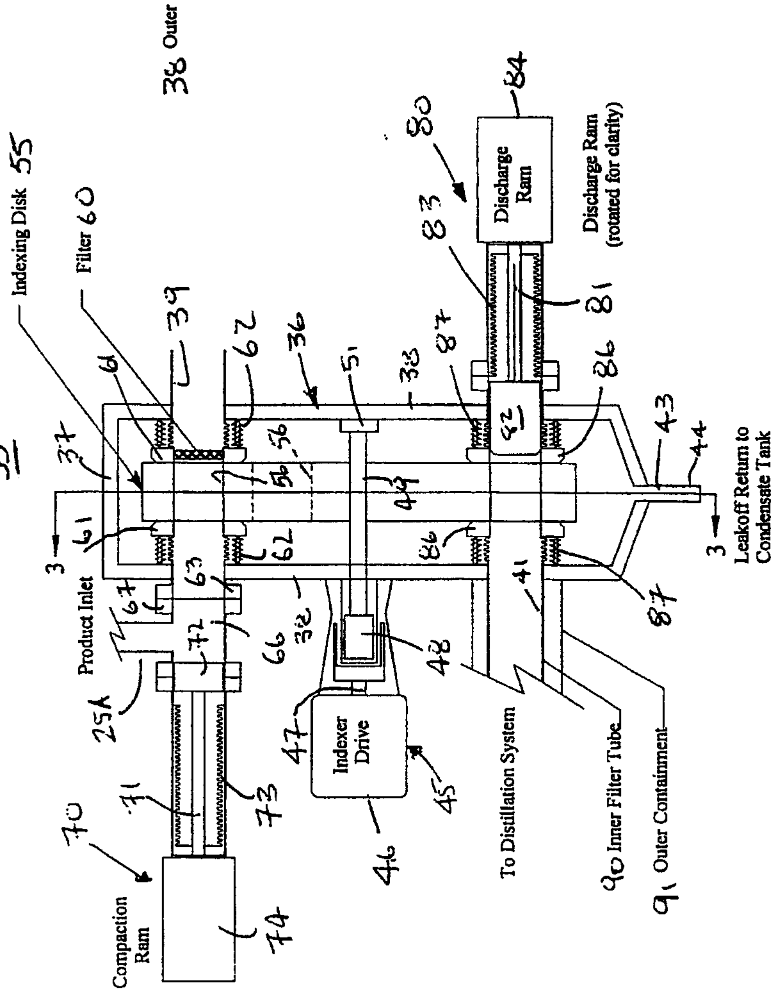


Figure 3

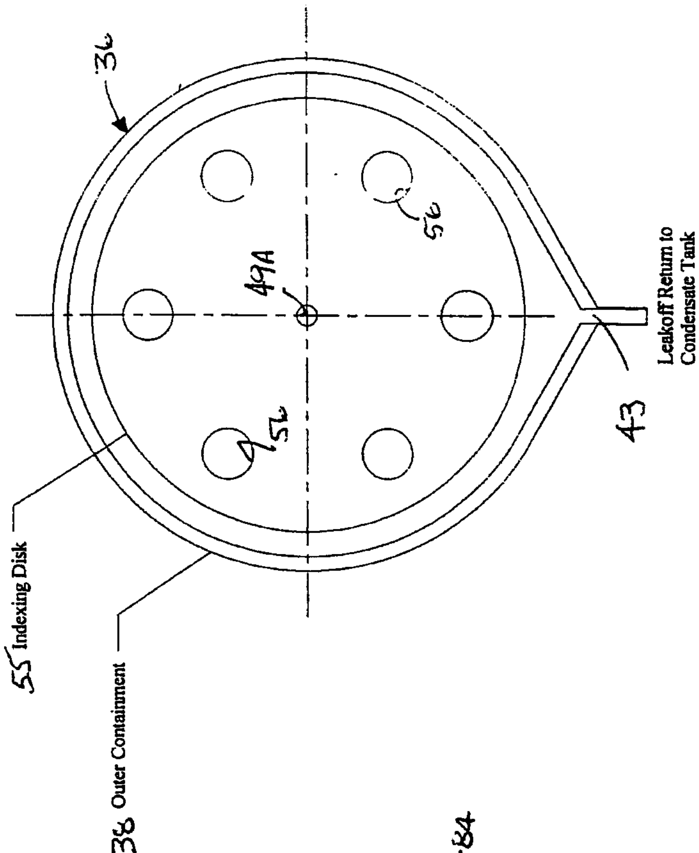
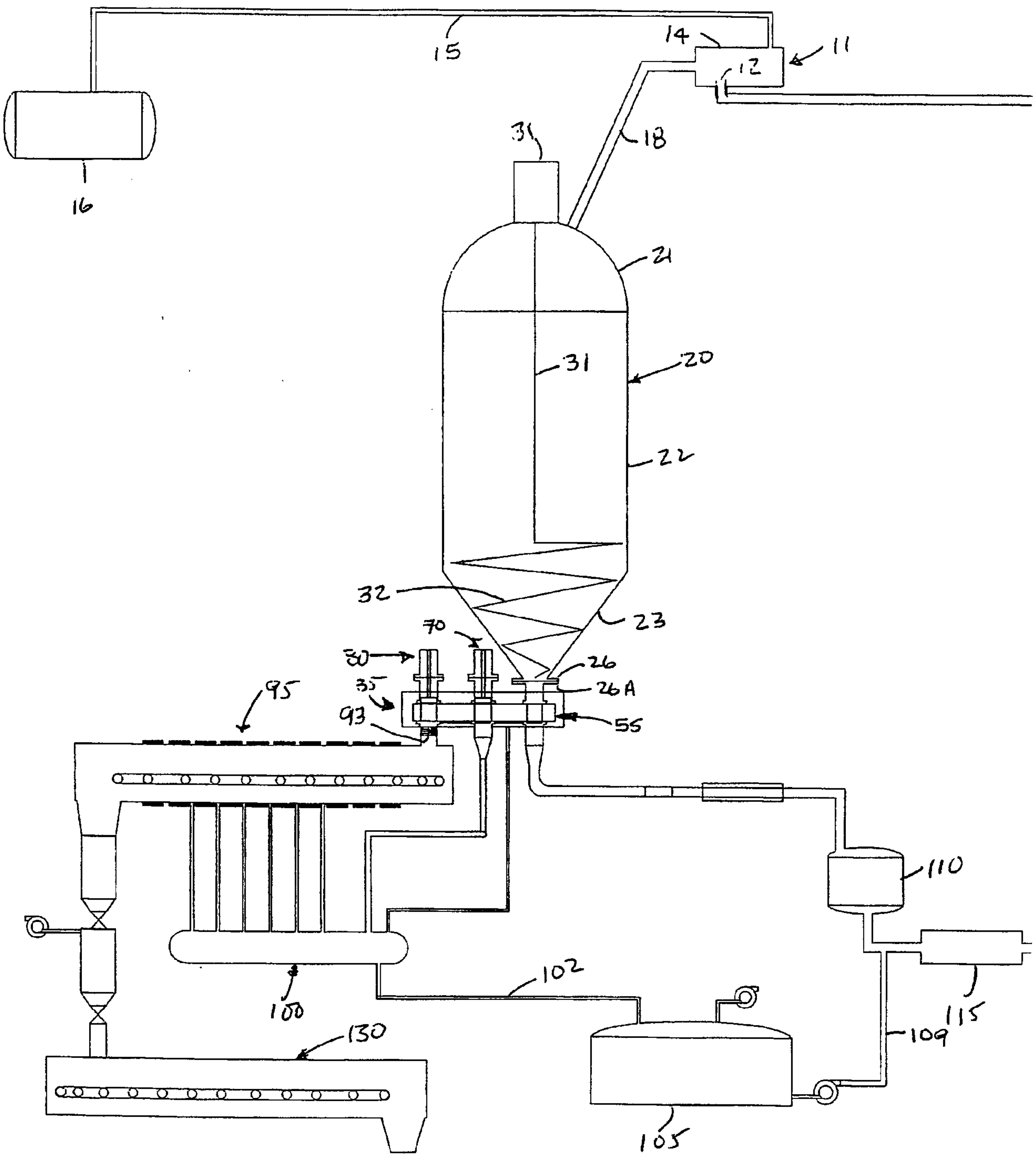


Figure 4



To Wash/Dry System

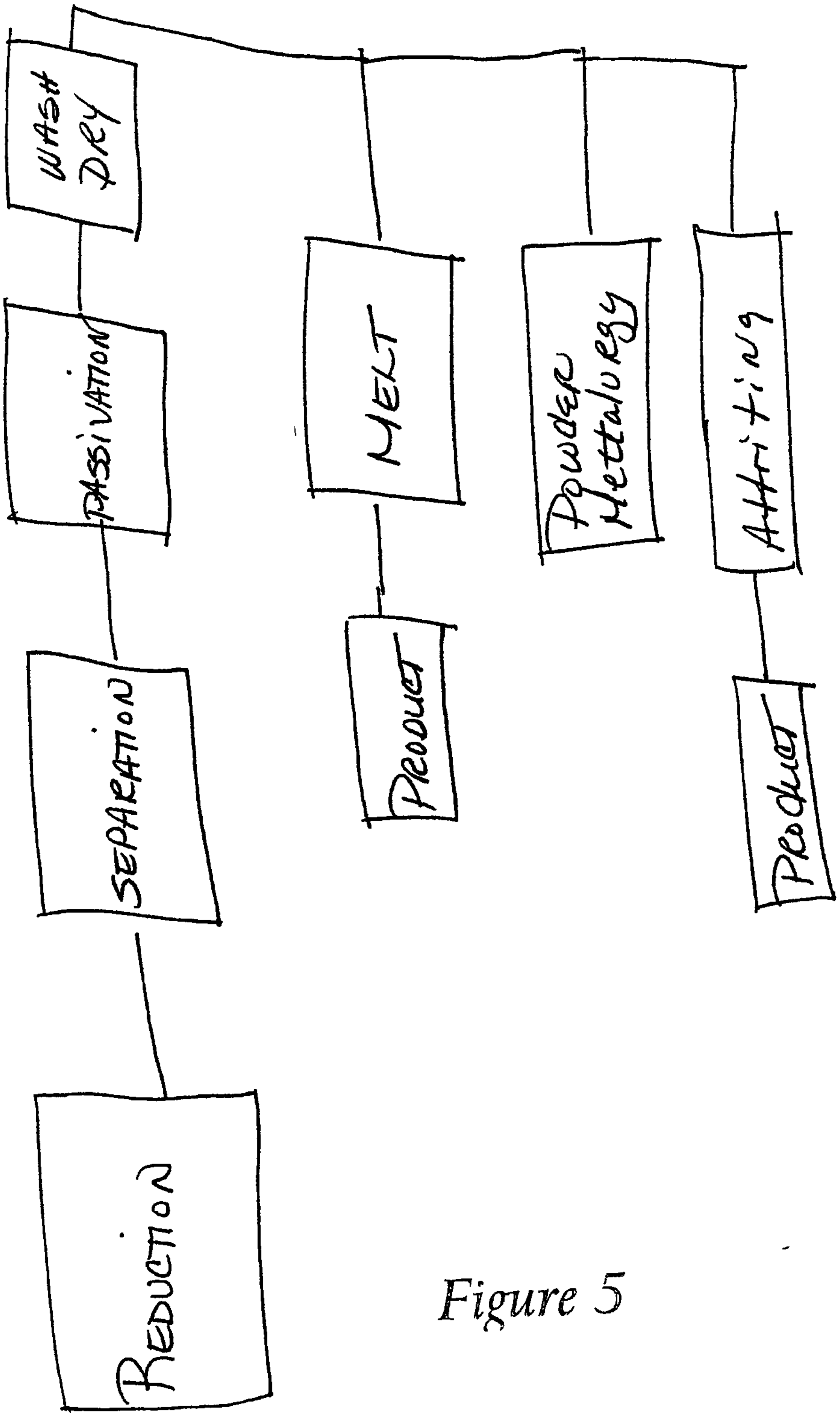

Reduction
Separation
Passivation
Wash
Dry
Melt
Product
Powder
Mettalurgy
Attriting
Product

Figure 5

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/027277

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C22B34/12 C22B9/02 B01D29/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C22B B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	sections 0022 and 0025; claim 4	1-68
X	US 2 882 143 A (SCHMIDT CHARLES L ET AL) 14 April 1959 (1959-04-14)	14-17
Y	column 2, lines 10-17; column 5, line 63 - column 6, line 15; column 9, lines 33-40	1-68
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Y	US 2003/145682 A1 (BORYS STANLEY S ET AL) 7 August 2003 (2003-08-07) Title	2,37

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
1 December 2004	30/12/2004

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Bjoerk, P

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/027277

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Y	LU L, FUH J.Y.H. AND WONG Y.S.: "Laser-induced materials and processes for rapid prototyping" 2001, KLUWER ACADEMIC PUBLISHERS , USA , XP002308397 pages 153-154	25
X,P	WO 2004/028655 A (ARMSTRONG DONN ; JACOBSEN LANCE (US); ANDERSON RICHARD (US); INTERNAT) 8 April 2004 (2004-04-08) Claims	1

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Information on patent family members

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